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[54] **FLANGE AND COUPLING COOLING MEANS AND METHOD**

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[51] **Int. Cl.**[3] **F27D 7/00**; F27D 5/00; F27D 1/12; F27B 14/10

[52] **U.S. Cl.** **432/1**; 65/DIG. 8; 219/405; 432/65; 432/237; 432/253; 432/262

[58] **Field of Search** 432/1, 32, 65, 253, 432/262, 237; 219/405; 422/240; 65/DIG. 8

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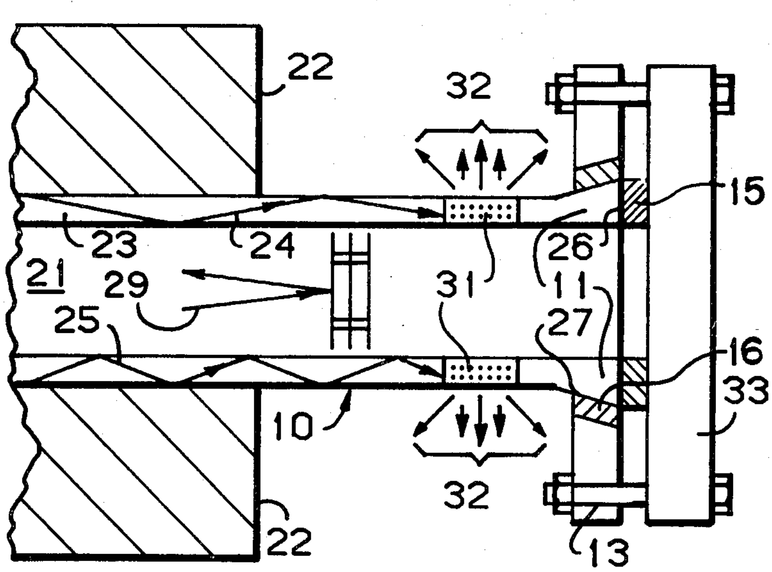
[57] **ABSTRACT**

Reaction vessels, furnace tubes, heating and cooling enclosures frequently have parts such as access ports, inlet tubes, inspection windows, etc. made of thermally transparent materials such as plastic, glass, quartz, oxides, nitrides and sulfides. When these parts extend outside the hot zone, they can act as "light pipes" carrying appreciable amounts of thermal radiation which can damage thermally sensitive gaskets or other materials used to secure external couplings or end closure flanges to these parts. Thermal radiation induced gasket damage is a frequent cause of stuck flanges and couplings. This problem is avoided by inserting a thermal radiation scattering region in the thermally transparent material between the hot zone and the end closure or gaskets. The thermal radiation is scattered and dispersed, so that the end zones receive less radiation and remain cooler. Milky quartz is a suitable scattering material for use with quartz furnace tubes or bell jars.

15 Claims, 3 Drawing Figures



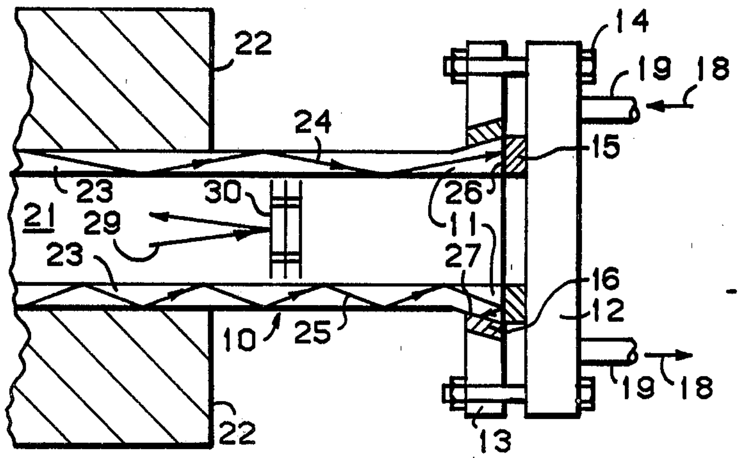


FIG 1
-PRIOR ART-

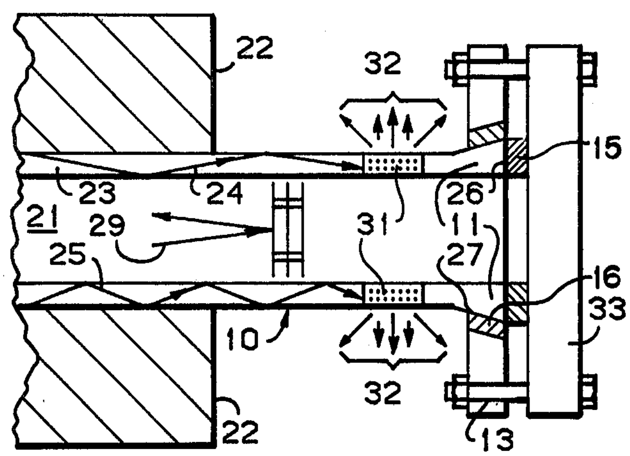


FIG 2

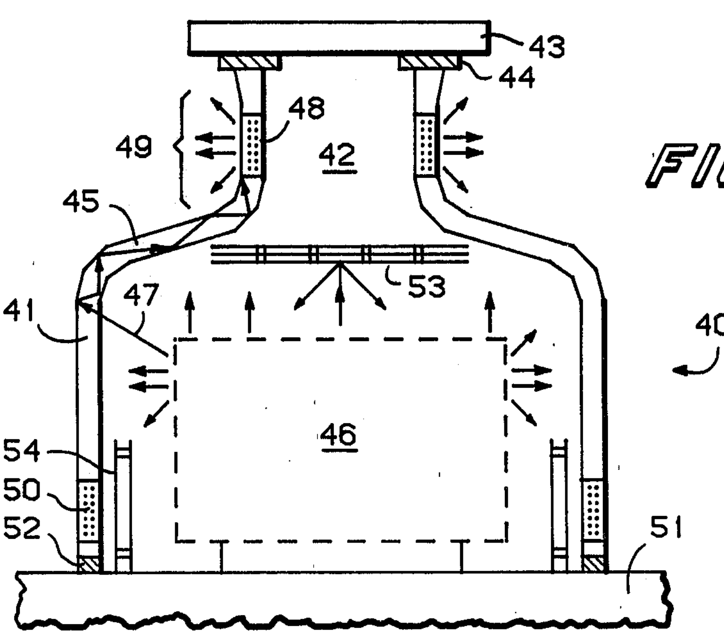


FIG 3

FLANGE AND COUPLING COOLING MEANS AND METHOD

BACKGROUND OF THE INVENTION

This invention relates to improved heating or cooling enclosures or reaction vessels having transparent protrusions, and more particularly, to means and method for reducing the thermal radiation being carried along such protrusions.

Glass, quartz, fused alumina and other optically and thermally transparent materials are widely used as enclosures for heating or cooling of materials. A vitreous quartz diffusion tube or bell jar for the processing of semiconductor materials is a typical example. In this example, the semiconductor materials are heated within a quartz tube in a furnace, epitaxial reactor, or other deposition apparatus. A portion of the quartz tube usually extends outside of the hot zone and is typically terminated by a removable coupling or flange which permits access or observation. Rubber, Teflon, Viton, other elastomers and other thermally sensitive materials are frequently used as gaskets on these flanges. If excess thermal energy is transmitted to these materials from the hot zone of the furnace or reactor they are damaged and stick to the flanges and tubes. It then becomes extremely difficult to separate the flanges from the tube ends without damaging the tubes or flanges or both. This has been found to occur even though the flanges and the extensions of the quartz tube beyond the hot zone are cooled by conductive or convective means. While placing a radiation reflector in the interior of the quartz tube between the hot zone and the flange reduces the amount of radiant heat coupled directly to the flange, it has not served to eliminate the problem of damage to the gaskets.

It has been discovered that a significant amount of radiant energy is coupled to the gasket or attachment areas by means of thermal and optical radiation being carried inside the wall regions of the quartz tubes. The wall regions of the quartz tube act as a "light pipe" carrying thermal radiation from the hot zone to the gaskets or attachment areas of the closure means. High intensity thermal radiation can be carried by the quartz material even though the local temperature of the quartz itself is relatively low. By this means, a large amount of energy can be coupled from the hot zone to the material of the gaskets or attachment areas, independent of the convective or conductive cooling means being applied to the quartz tube or the end flange.

This "light pipe" effect can be easily demonstrated by placing a quartz tube or rod in a high temperature flame. The portion of the rod a short distance outside the flame is cool to the touch, but if one's hand is placed facing the end, the heat being transmitted (carried) along the rod can be readily felt. Thermal radiation burns can result. For the purposes of this disclosure a thermal radiation carrying member, media, or enclosure is one which exhibits this light pipe effect, that is, channeling or carrying thermal radiation by radiant propagation from a hot zone to other regions.

Thus, a need continues to exist for reducing the coupling of radiant energy from hot zones to exterior couplings or flanges of transparent reactor enclosures, this radiation being carried in the walls of the enclosure itself.

Accordingly, it is an object of this invention to provide an improved apparatus for protecting closure means of a radiation carrying member coupled to a hot zone, from the radiant thermal energy being carried by said member between said hot zone and said closure means.

It is a further object of this invention to provide an improved apparatus for protecting a closure flange of a high temperature semiconductor reaction chamber from radiation being piped in the walls of the chamber.

It is an additional object of this invention to provide an improved process for heating materials in an enclosure of which a wall portion channels the thermal radiation from a source region to a closure region, said closure region being susceptible to damage by said radiation.

It is a further object of this invention to provide an improved method for protecting a closure means of a thermal radiation carrying member from the radiation being carried by the material of said member.

It is an additional object of this invention to provide an improved method for heating semiconductor materials to high temperatures in an enclosure while maintaining a coupling or closure flange connected thereto at a lower temperature.

It is a further object of this invention to provide an improved method for cooling a coupling or closure means of a semiconductor materials processing enclosure.

SUMMARY OF THE INVENTION

The attainment of the foregoing and other objectives and advantages is achieved through the present invention wherein the radiation path in the walls of a radiation carrying member portion of a reaction chamber containing a radiation source (not zone) is interrupted by placing a radiation scattering region in the material of the member portion serially disposed between the hot zone and the closure means. This radiation scattering region intercepts and disperses a portion of the radiant energy being carried in the walls of the member portion and thus prevents it from reaching the closure means. Elastomer gaskets or other temperature sensitive materials in the closure means are thereby substantially protected from this source of energy.

BRIEF DESCRIPTION OF THE DRAWINGS

A more complete understanding of the present invention can be obtained by considering the following detailed description in conjunction with the accompanying drawings in which:

FIG. 1 illustrates a cross section of a prior art example of a quartz reaction tube extending from a hot zone of a furnace and having an external cooled closure flange;

FIG. 2 illustrates a reaction tube similar to FIG. 1 but with the radiation scattering zone of the present invention installed between the hot zone and the closure flange; and

FIG. 3 illustrates a cross section of a bell jar reaction chamber utilizing the present invention to reduce the heat input to a closure flange.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

It is useful to briefly discuss the mechanisms by which hot bodies give off energy and to define a number of the terms used in the present application.

A hot object or body radiates electromagnetic energy at all wavelengths. However, for most industrial processes the amount of energy being radiated at very short or very long wavelengths is relatively insignificant, and the majority of the energy is typically radiated in a band of wavelenths extending from the far infrared through the visible spectrum. The term "thermal radiation" as used herein refers to the electromagnetic radiation being given off by a hot object or media irrespective of whether that radiation is visible or invisible to the human eye. The wavelength regions of principal interest extend from the far infrared (1000 microns) to the ultraviolet (less than 0.4 micron). As the temperature of the hot body increases, the wavelength associated with the maximum radiant energy output decreases. Thermal radiation is an electromagnetic phenomena encompassing both "heat" and "light" and is physically distinct from ordinary thermal conduction or convection which proceeds by different mechanisms. As used herein, "high temperature", "hot", and "hot zone" are relative terms denoting a significant temperature difference and not necessarily a large absolute temperature.

Thermal radiation propagates in vacuum and in transparent media. A transparent media is one which passes, transmits or carries electromagnetic radiation of a given wavelength without significant scattering or absorption. Common examples of transparent media are plastic, glass, fused (vitreous) or crystalline quartz, oxides, nitrides, sulfides, and combinations thereof, but many others exist. Transparency need not be perfect. Information on the optical transparency of various media can be found, for example, in the "*Modern Plastics Encyclopedia*", published annually by McGraw Hill, New York; in the "*Handbook of Physics and Chemistry*", published bi-annually by CRC Press, Inc., Boca Raton, Fla.; in the "*Handbook of Electronic Materials*", IFI/Plenum, New York 1971; in the "*Properties of Glass*", by G. W. Mosey, Reinhold N.Y., 1954; in the "*Introduction to Ceramics*", by W. D. Kingery et al, John Wiley and Sons, New York, 1976; and in other handbooks and other texts well-known in the art.

Once thermal radiation has entered a transparent media, it can become trapped within the media because of the difference in dielectric constant of the media relative to the external ambient. Radiation rays within a relatively thin media are reflected back and forth from the interior surfaces so that the media acts as a "light pipe" or conduit channeling thermal radiation from one region or portion to another. Bends, angles, or directional changes in the material do not impede the light pipe effect when the radius of curvature of these directional changes is relatively large compared to the thickness of the material, so that the rays within the material can continue to make mostly low angle reflections from the internal surfaces.

Thus, thermal radiation entering the walls of a quartz vessel, for example, can be carried along within the walls to portions extending outside of the hot zone until the walls terminate or encounter an end means, coupling, closure means or flange. At this point, the propagating rays of thermal energy encounter an end or other surface with new materials of different dielectric constant or strike at a high angle or both, and exit without difficulty. If an elastomer or other temperature sensitive material is present against this end face or other surface, the thermal energy will be coupled directly to that material. The effective temperature of this radiant energy can be much higher than the actual local temperature of the container wall material (e.g. the quartz) through which it is being conducted. Thus, damage to temperature sensitive closure materials can easily result. As used herein, the words "end means", "closure means", "coupling" or "flange" are intended to be synonomous and refer to any arrangement, mechanical or otherwise, whereby a thermal radiation carrying member is closed-off or connected to another body, media, or member.

FIG. 1 illustrates a prior art example of a quartz reaction tube **10** having an end region **11** to which is attached flange **12** and flange collar **13** by bolts **14** together with elastomer gaskets **15–16**. Quartz reaction tube **10** extends from hot zone **21** of furnace **22**. Thermal radiation originating within hot zone **21** enters wall portion **23** of quartz tube **10**. Thermal radiation rays **24–25** within wall portion **23** propagate from hot zone region **21** toward end region **11** where they intersect end surfaces **26–27** and are absorbed by elastomer gaskets or other thermally sensitive materials **15–16**. Additional thermal radiation ray **29** originating within hot zone **21** and propagating down the interior of quartz reactor tube **10** is intercepted by radiation baffle **30**. Cooling means **18–19** can be used to remove heat from flange **12** so as to cool one face of gasket **15**. However, it has been found that one or both gaskets **15–16** continue to suffer thermal radiation damage.

FIG. 2 illustrates a tube similar to FIG. 1 but with radiation scattering zone **31** installed between hot zone **21** and flange **33** so that radiation rays **24–25** are intercepted and scattered in other directions as illustrated by rays **32**, so as to significantly reduce the radiant energy reaching faces **26–27** of gaskets **15–16**. The scattering zone is thus serially disposed in the radiation pathway or channel between the hot zone (radiation source) and the closure means so as to intercept and disperse radiant energy propagating in the channel. The method of forming scattering zone **31** will be explained subsequently.

FIG. 3 illustrates alternative embodiment **40** in which bell jar reaction chamber **41** has radiation baffles **53–54**, neck region **42**, and closure means **43** with gasket **44**. Within chamber **41**, hot zone **46** radiates thermal energy **47** into wall region **45** of bell jar **41**. Thermal radiant energy **47** is intercepted by scattering zone **48** and dispersed as rays **49**. Similarly, scattering zone **50** can intercept and disperse radiant thermal energy being piped toward gasket **52** on base plate **51**.

As an example of the use of the present invention, a substantially transparent vitreous quartz enclosure of the type illustrated in FIG. 3 but without radiation scattering region **48** was sealed with O-rings to flange **43**. With a hot zone temperature of 1200° C., it was found that the O-rings reached temperatures of 300° C. This temperature of 300° C. is sufficient to cause the O-ring seals to stick to flange **43** and neck region **42** of the quartz vessel. It was frequently necessary to fracture the quartz vessel in order to remove the flange. This problem occurred even when forced convection or other cooling was provided for neck region **42** or flange **43**. When the same structure was used under the same conditions but with thermal radiation scattering region **48** installed, the temperature of the O-rings remained below 200° C. No thermal damage or sticking was encountered, and no forced cooling was needed.

In the above experiment, neck region **42** was formed from a standard four inch (10 cm) interior diameter quartz pipe with a $\frac{3}{8}$ inch (1 cm) wall thickness and $\frac{3}{4}$

inch (2 cm) end face contacting O-ring gasket **44**. Flange **43** was metal. Thermal radiation scattering region **48** was varied in length from two to six inches (5–15 cm). While a two inch (5 cm) length of scattering region provided some improvement, greater blockage of piped thermal radiation was obtained using four to six inches (10–15 cm). Four inches (10 cm) was adequate to eliminate sticking even when placed directly in contact with gasket **44**. The scattering region was formed of high purity crystalline quartz sand fused at a temperature sufficiently high to bond together the grains of the sand but not destroy their individual crystallinity. As a consequence, this material, while having substantially the same chemical and mechanical properties as clear fused quartz, has a milky appearance, is nearly opaque, and is an effective radiation scattering media. Because the coefficients of expansion are substantially the same as clear quartz, it can be readily sealed to quartz reaction vessels without encountering undesirable thermal stress problems. This milky form of quartz is a commercially available material well known in the art. An example is "opaque quartz" sold by the Thermal American Fused Quartz Company of Montville, N.J.

While a specific embodiment of the invention has been demonstrated using clear fused quartz as the reaction vessel material and milky fused quartz as the thermal radiation scattering region material, it will be readily apparent to those skilled in the art that the invented concept can be used with other material combinations, for example plastics, glass materials, oxides, nitrides or sulfides or combinations which are substantially transparent to thermal radiation in the wavelength region of interest for the particular source temperature being used. Data on the optical properties as a function of wavelength of various oxides, nitrides, sulfides and other materials can be found, for example, in the "Handbook of Electronic Materials", IFI/Plenum, New York 1971. See especially Volumes 1 and 3.

While it is desirable that the radiation scattering region be made of the same material as the reaction vessel but having scattering centers dispersed within so as to alter the optical properties while maintaining substantially the same mechanical and chemical properties, this is not essential and other material combinations will serve. It is desirable to minimize the mechanical stress which may result from differential thermal contraction of materials of different expansion coefficients.

Materials having desirable thermal radiation scattering properties may be formed by various sintering and melting procedures, as with the illustrated example of milky quartz, or by entrainment of air bubbles or foreign material, or by nuclear irradiation damage, or by other methods which produce a high density of localized radiation scattering sites.

The extent of the thermal radiation scattering region required to give effective dispersement of the thermal radiation being carried in the walls of the vessel or member is dependent upon the volume density of scattering sites and can be readily determined by experiment. The higher the volume density of scattering sites, the shorter may be the scattering region.

Thus, it is apparent that there has been provided in accordance with this invention an improved structure and method for protecting the closure or coupling means of a radiation carrying member from the radiant thermal energy being carried by the walls of that member from a hot zone. Further, it is apparent that there has been provided an apparatus and a method for the improved heating of materials, particularly semiconductor materials, to high temperatures in an enclosure while maintaining the end faces or other surfaces of that enclosure at a lower temperature than has previously been possible. It is further apparent that there has been provided in accordance with this invention a means for eliminating the forced cooling of couplings or end flanges on enclosure means of transparent reactor vessels capable of light pipe conduction of radiant energy.

Having thus described the invention, it will be apparent to those of skill in the art that various modifications can be made within the spirit and scope of the present invention. For example, this invention may be used to reduce thermal radiation being coupled into a low temperature cryostat along transparent members. Accordingly, it is intended to encompass all such modifications.

We claim:

1. An enclosure in which heating or cooling occurs, comprising:

a vessel having a region adapted to include a hot zone;

a thermal radiation carrying member composed of a predetermined material and coupled to said vessel, extending outside said region, and able to receive thermal radiation from said hot zone;

end means coupled to said member outside said region; and

thermal radiation scattering means integral with said member and serially disposed between said region and said end means, so that a portion of said thermal radiation originating in said hot zone and carried within said predetermined material of said member is prevented from reaching said end means.

2. The enclosure of claim **1** wherein said scattering means comprises a radiation scattering material region of substantially the same thermal coefficient of expansion as said predetermined material.

3. The enclosure of claim **1** or **2** wherein said predetermined material is selected from the group consisting of substantially transparent nitrides, oxides, and sulfides.

4. The enclosure of claim **1** wherein said predetermined material is vitreous quartz and said radiation scattering means is composed of substantially opaque milky quartz.

5. In a thermal radiation carrying member composed of a predetermined material, extending from a zone able to give off thermal radiation, and having an end means, the improvement comprising:

a thermal radiation scattering region installed in said material of said member between said zone and said end means, so that a portion of said radiation being carried in said member through said material is dispersed before reaching said end means.

6. The improvement of claim **5** wherein said material of said member is selected from the group consisting of glass and vitreous quartz.

7. The improvement of claim **5** wherein the material of said member is oxide, nitride, sulfide, or combinations thereof.

8. The improvement of claim **6** wherein said radiation scattering region is a milky form of said material.

9. The improvement of claim **6** wherein said scattering region has substantially the same thermal coefficient of expansion as said member.

10. The improvement of claim **9** wherein said member comprises a means for enclosing, at least in part, said hot zone.

11. The improvement of claim 9 wherein said member comprises a means for accessing said hot zone.

12. The improvement of claim 7 wherein said scattering region has substantially the same thermal coefficient of expansion as said member.

13. The improvement of claim 12 wherein said member comprises a means for enclosing, at least in part, said hot zone.

14. The improvement of claim 12 wherein said member comprises a means for accessing said hot zone.

15. A method for protecting a closure means for a thermal radiation carrying member composed of a predetermined material, from radiation being carried by said member within said material from a portion of said member exposed to a thermal radiation source, comprising:

installing serially in said material of said member a thermal radiation scattering zone between said source and said closure means so as to reduce the thermal radiation reaching said closure means.

* * * * *